The newer phenolic and vinyl base paints present less of a health hazard from lead intoxication than oil base paint, according to this study which investigates the effect of a number of variables on lead solubility.

Laboratory Study of the Solubility of Red Lead Paint in Water

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CONCERN increases over the amount of lead taken into the body as it becomes more and more apparent from analyses of food, air, and water that each of these contributes a quantity of lead each day. The maximal non-cumulative daily body burden of lead has been determined to be approximately 0.5 mg. Lead intake from sources other than water approaches this permissible maximum. If it is found that any one of the three sources contributes an undue share of lead, efforts should be made to identify that source and suggest a remedy.

In view of the extensive industrial use of red lead and red lead paints and the hygienic significance of lead compounds, it is remarkable that so very little is known about the solubility of these materials in water. According to Friend (1), red lead is "practically insoluble in water." The only value we have found in

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the literature is one theoretically derived by Glasstone (2) from electrode potential measurements. Glasstone measured the electrode potential of the half element $Pt\begin{bmatrix} Pb_3O_4\\ PbO\end{bmatrix}$ against the standard mercury-mercurous oxide electrode using identical solutions of carbonate-free-N-sodium hydroxide throughout. The value obtained of -0.610 is very slightly higher than the theoretical potential of -0.617 volt. Assuming the ionization of red lead to be $Pb_3O_4 \rightleftharpoons 2 Pb^{++} + PbO_4^{----}$ the solubility of red lead in N-NaOH is 1.1×10^{-17} gram-mol per liter at 17° C.

Our study was undertaken to determine experimentally the solubility of red lead and the extent of dissolution of lead by water in contact with surfaces coated with red lead paint. Three types of red lead paint were tested: linseed oil base, phenolic base, and vinyl base. Specifications of these paints are given on p. 502. It should be noted that the oil base paint used did not contain the added litharge found in presentday specifications. Several lots of oil base paint, some ground commercially and some in the laboratory, were studied. In a supplementary experiment the three types of red lead paint were exposed to the solvating action of natural and treated waters from eight localities in the United States.

Since the data were obtained in the laboratory

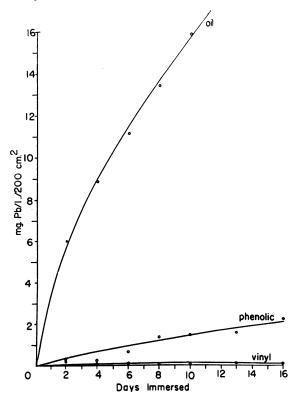
and selected variables affecting the solubility of lead were studied separately, the study does not apply to all variables, acting in combination, that may be encountered in the field. For a valid toxicological appraisal of the amounts of lead in water, samples taken from tanks actually in use with various types of water would have to be analyzed.

Red Lead Powder

The chemical procedures for determining the amount of lead taken up by water in contact with red lead may be interfered with by (a) the particle size of the red lead, (b) the colloidal tendency and penetration through filtering media, and (c) adsorption of lead by the filtering medium.

Measurements of the particle size of the samples of red lead powder showed an average of 0.8 micron (μ) , with a range from 1.3μ to the lower limit of visibility of the optical micro-

Figure 1. Dissolution of lead from red lead paint in contact with distilled water.



Note: Drying time for oil base paint: 80 days.

Specifications and Formulas for Paints Tested

Oil Base

From Federal Standard Stock Catalog, Section IV, Part 5. Federal Specification for Paint; Red Lead Base, Ready Mixed, TT-P-86a (May 4, 1949), type I.

I	Percent
Component by	veight
Red lead (Fed. Spec. TT-R-191, type I,	
grade C)	77.53
Aluminum stearate (Navy Spec. 52 A 12)	. 23
Raw linseed oil (Fed. Spec. TT-O-369)	10.90
Pale heat-bodied linseed oil (Navy Spec.	
52020)	3.78
Mineral spirits (Fed. Spec. TT-T-291, grade	
1)	6.45
Liquid drier (Fed. Spec. TT-D-651, type I)_	1. 11
-	100.00

Phenolic Base

From Federal Standard Stock Catalog, Section IV, Part 5: Federal Specification for Paint; Red Lead Base, Ready Mixed, TT-P-86a (May 4, 1949), type IV.

	Percent
Component by	y weight
Red lead (TT-R-191, type I, grade C)	55.99
Magnesium silicate (TT-M-90)	4. 36
Diatomaceous silica (52-MC-522, type I)	5. 25
Aluminum stearate (52 A 12)	. 26
Phenolic varnish	25.35
Aromatic petroleum spirits (TT-N-97, type	
II)	7. 60
Diptene (TT-D-376)	. 94
Lead naphthenate (24 percent Pb)	. 08
Cobalt naphthenate (6 percent Co)	. 02
Manganese naphthenate (6 percent Mn)	. 02
Antiskinning agent (National Aniline ASA)	. 13
•	100.00

Vinyl Base

No Federal specification available. Red lead vinyl resin paint, supplied by National Lead Co., Brooklyn, N.Y.

	Percent
Component	y weight
Red lead (TT-R-191, type I, grade C)	23.70
Aluminum stearate	. 10
Vinyl resin—VAGH	. 16.35
Tricresyl phosphate	. 1.64
Toluene	25. 31
Methyl isobutyl ketone	25.31
Carbitol (low gravity)	7. 59
	100.00

scope, about 0.5μ A suspension showed active Brownian movement, and the colloidal aggregates precluded filtration through even the best grade filter paper. A certain amount of colloidal lead invariably penetrated the filter, as shown by the Tyndall effect, with resultant erroneously high values.

The adsorption of lead on the filter paper causes erroneously low values. O'Shea (3), who first demonstrated the adsorption of lead by filter paper, showed that filtration of dilute lead acetate solution resulted in a loss of approximately 0.2 mg. of lead in 50 cc. of solution. Preliminary experiments in this study showed that filter paper adsorbed lead from neutral solution to such an extent that three filtrations of an optically clear solution of 1 mg. of lead salt per liter of water removed 99 percent of the lead.

Ordinary filtration procedure was therefore excluded, and pressure filtration through cellophane with the Zsigmondy type of apparatus was resorted to. When wettable cellophane, designated as 600–PUT–O, was used as the filtering medium under a pressure of 150 kg./cm.²

Figure 2. Effect of drying time on dissolution of lead from oil base red lead paint.

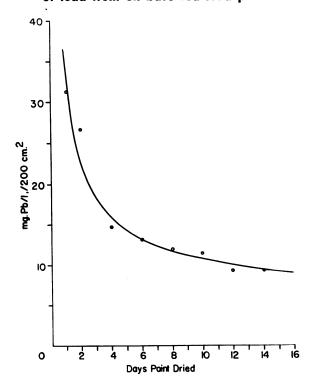
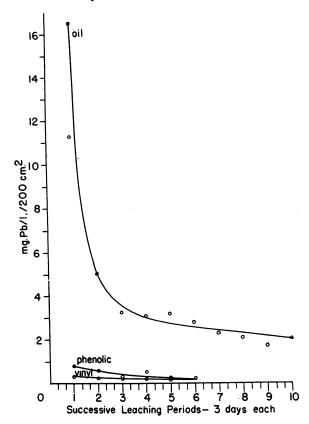


Figure 3. Effect of successive leaching of red lead paint film with water.



Note: Drying time for oil base paint: 10 days.

of nitrogen gas, no detectable lead loss occurred and the filtrates were optically clear.

Suspensions of red lead in distilled water were shaken in Pyrex bottles until equilibrium was reached. The lead content of the pressure-filtered solutions was determined by the chromate method of Fairhall and Keenan (4). This analytical method was chosen because, being a titration method, it is applicable over a wide range of concentrations without dilution of the sample or other extrapolation of results.

Because of probable contamination of red lead with the more soluble lead monoxide, reagent-quality red lead was purified by Glasstone's method by repeated treatment with sodium hydroxide (1), and then washed with dilute acetic acid. The successive portions were analyzed for lead until constant values were obtained.

Purified reagent grade red lead powder gave an average value in distilled water at room temperature of 5.53 x 10⁻⁴ gram of lead per liter (0.553 ppm) or 8.9 x 10⁻⁷ gram-mol of Pb₃O₄ per liter. Suspensions of red lead in distilled water gave only a slightly greater amount of lead in true solution after standing for 1 year.

Red Lead Paint

The investigation of red lead paint was concerned solely with the lead in solution. Particulate lead oxide or particles of lead paint film resulting from blisters or pinholes and mechanically swept into suspension, while hygienically important, were completely removed, and the results in all cases refer only to lead in true solution.

Although the effect of water alone was of primary interest, the effects on lead solubility of sodium hydroxide, sodium hexametaphosphate, free chlorine, and differences in alkalinity and acidity were also studied.

For most of the tests the paint was applied

to glass surfaces. Glass was used so that the results would reflect the solution tendency of the paint film itself rather than the effect of possible interaction of the paint film and metal surfaces. Several dozen glass plates were cut to measure 3 inches by 6 inches from stock 0.3 cm. thick. Areas of 100 cm.2 were measured and marked off on each plate. The plates were washed, cleaned with nitric acid and distilled water, dried, and painted to the desired area, and the paint film was allowed to dry for appropriate periods. In studying the oil base paint, various drying times were tested. The drying time of the phenolic and vinyl base paints was less important, since drying consists principally in evaporation of the solvents (5).

Water solubility tests were also made for lead from the three paints on black iron and on galvanized iron plates. The red lead paint was applied directly to the iron plates or over an undercoating of zinc-chromate primer.

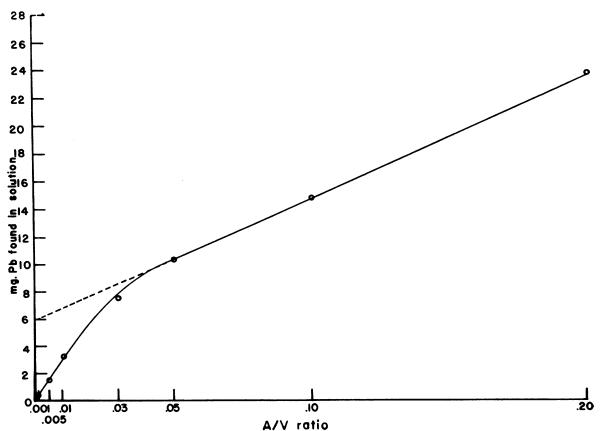
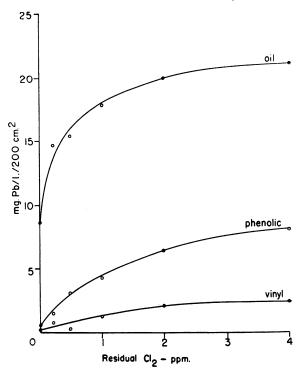


Figure 4. The amount of lead in solution plotted against the area/volume ratio (cm.2/cm.3).

Note: Data for oil base paint, with drying time of 7 days and immersion time of 3 days.

Figure 5. Effect of chlorination of water on dissolution of lead from red lead paint.



Note: Drying time for oil base paint: 94 days. Immersion time: 3 days.

When the primer was used, the red lead paint was followed by a coat of aluminum paint.

The painted plates, both glass and metal, were placed in tall form 1-liter beakers, and 500 ml. of either distilled water or the desired salt solution was added. The beakers were sealed with sheet Parafilm and allowed to stand at room temperature for the desired time. The plates were then removed, rinsed with distilled water, and the water was analyzed for lead by the chromate method (4).

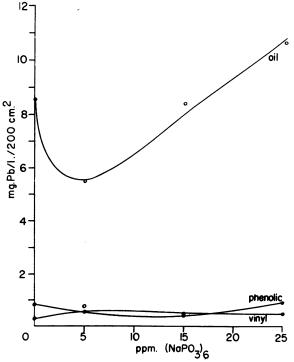
Finally, an experiment was performed in which successively smaller areas were painted with the oil base paint and dried for 7 days. These glass plates were immersed in successively greater volumes of water. After immersion for 30 days the water was drained off and tested for dissolved lead by the more sensitive dithizone method (6). This procedure permitted an investigation of the range of area/volume ratios from the experimentally determined ratios to those that might be encountered in the field.

In the supplementary investigation the three types of red lead paint were exposed to natural water. To determine the effect of variations in natural water on the solution of lead, samples of the treated city water supplies from eight localities in the United States were obtained. Areas measuring 100 cm.² on glass plates were painted with the red lead paint, and the plates were immersed in 500 ml. of each of the water samples for 30 days after the paint had dried for 7 days. The water was then analyzed for lead content by the chromate method (4).

The results of all experiments are expressed in milligrams of lead dissolved per liter of water for exposure surface of paint film of 200 cm.² In all experiments except the area/volume ratio study, the area/volume ratio was 100 cm.²/500 ml., or 1 cm.² of surface per 5 ml. of solution.

Immersion time. The results of the immersion-time experiment show clearly that the oil base paint yields increasing amounts of lead with continued exposure (fig. 1). After 10

Figure 6. Effect of sodium hexametaphosphate on dissolution of lead from red lead paint.



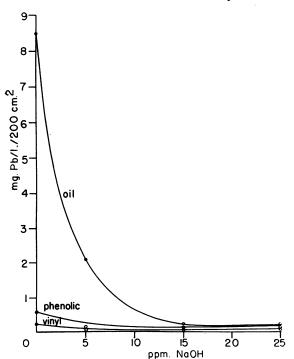
Note: Drying time for oil base paint: 97 days. Immersion time: 3 days.

days' immersion, the lead content of the test water was 15.8 mg. per liter. Furthermore, the oil base paint yields more lead in water than either of the other two paints. The highest value for the phenolic base paint was 2.32 mg. The highest for the vinyl base paint, which yielded the least amount of lead of any of the paints, was 0.13 mg.

Drying time. For the oil base paint, increasing the drying time from 1 day to 2 weeks appreciably decreased the release of lead, from 31.24 mg. to 9.30 mg. after 3 days' immersion time (fig. 2). Drying beyond the 2-week period caused a further slight reduction. However, an average value of 7.44 mg. per liter of water was obtained after 80 days of drying. Since the drying of the phenolic and vinyl paints consists largely in evaporation of solvent rather than the complicated chemical reactions which occur in the drying of linseed oil base lead paint, no particular change in the amount of lead in solution would be anticipated, and none was observed.

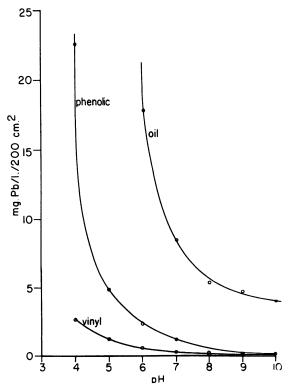
Repeated immersion. Repeated immersions,

Figure 7. Effect of sodium hydroxide on dissolution of lead from red lead paint.



Note: Drying time for oil base paint: 97 days. Immersion time: 3 days.

Figure 8. Effect of variation in hydrogen ion concentration on dissolution of lead from red lead paint.



NOTE: Drying time for oil base paint: 14 days. Immersion time: 3 days.

each lasting 3 days, resulted, for the oil base paint, in a decided decrease from the first high value of 16.50 mg. of lead per liter of water until a nearly constant value of 2 mg. was obtained after the 10th immersion (fig. 3). A slight decrease was also noted for the phenolic and vinyl paints, but the initial solubility was much less. The decrease was from 0.81 to 0.21 mg. of lead per liter with the former and from 0.28 to 0.19 mg. with the latter.

Area/volume ratio. As the area/volume ratio (cm.²/cm.³) decreases from 0.2 to 0.05, a straight line relationship holds (fig. 4.). A further decrease results in values which lie considerably below those predicted by an extension of the straight portion of the curve.

Residual chlorine. Because public water supplies generally are chlorinated, the effect of residual chlorine on red lead was studied. With all three types of paint, lead solvency was increased by the presence of 0.0-0.2 ppm of

residual chlorine, the normal range of chlorination. With higher residual chlorine concentrations there was a further increase to a constant maximum. With the oil base paint the amount of lead dissolved in 3 days increased from a value of 8.54 mg. per liter per 200 cm.² in chlorine-free water to 14.54 mg. at a chlorine value of 0.2 ppm. Phenolic base paint under the same conditions increased from 0.59 to 1.62 mg., while vinyl base paint increased from 0.24 to 0.85 mg. (fig. 5).

Sodium hexametaphosphate. Addition to the water of low concentrations of sodium hexametaphosphate, another chemical often used in treating public water supplies, caused depression in the solubility of lead during an immersion period of 3 days (fig. 6). This effect has been noted previously by Ruchhoft and Kachmar (7) with reference to other lead salts. The solvent action of sodium hexametaphosphate was apparent only at relatively high concentrations (25 ppm). At the concentrations investigated this salt had little or no solvent effect on vinyl base red lead paint.

Sodium hydroxide. With increasing amounts of sodium hydroxide (up to 25 ppm) the amount of lead carried into solution decreased, for all three types of paint (fig. 7). With the oil base paint there was a decrease from 8.54 mg. after 3 days' immersion in distilled water to 0.23 mg. following immersion for the same period in water containing 25 ppm of NaOH.

Hydrogen-ion concentration. Variation in

pH from 4.0 to 10.0 indicated that water on the acid side of neutral has a greater solvent effect on paint film than does water on the alkaline side (fig. 8). Sodium acetate buffer with either acetic acid or sodium hydroxide was used to cover this entire range. The solution was made as weak as possible, consistent with obtaining buffering capacity, in order to limit the effect of the acetate ion and to record only the effect of the hydrogen-ion concentration. Immersion time was 3 days, and the oil base paint film was dried for 14 days prior to testing.

With all three paints, pronounced solubility of the red lead occurs at a pH of 4. The solubility value for oil base paint increased from 8.44 to 117.2 mg. per liter per 200 cm.² at this pH; for phenolic base paint it rose from 1.21 to 22.4 mg.; and for vinyl base paint, from 0.31 to 2.71 mg.

Nature of surface. Whereas most of the foregoing work was based on paint films on glass, further studies were made, as noted above, of red lead painted plates of black iron, galvanized iron, and iron plates with a priming coat of zinc chromate conditioner. When the iron plates were completely covered with red lead paint, the results were similar to those obtained with glass plates. The values were somewhat lower when the red lead paint blistered, formed pinholes, or became detached in any way so that the more basic metal was in contact with the solution. It was assumed that such decrease was due to the replacement of Pb ions by the metal higher in the E.M.F. series. Unpainted

Table 1. Characteristics of water in eight localities: Results of analyses supplied by local water departments

Locality pI		Parts per million											
	pН	pH Total solids	Na and K	HCO_3	SO4	Cl	$ m NO_3$	Hard- ness as CaCO ₃	Mg	Ca	Fe	SiO ₂	F
Amarillo, Tex El Paso, Tex Jacksonville, Fla Cleveland, Ohio Boston, Mass Catskill, N.Y Croton-on-the-Hud-	7. 90 8. 20 7. 95 7. 60 6. 65 7. 30	400 634 384 159 28 28	29 162 13 5. 6 3. 2 1. 7	326 24 150 113 7 11	36 265 150 25 6 8	14 121 20 11 2. 1 1. 0	2. 7 . 4 . 01 (¹) . 22 . 49	263 196 290 120 15 20	39 19 32 8. 4 . 7 1. 2	41 34 70 35 3 4. 5	0. 02 . 06 . 06 . 07 . 02 . 04	70 13 23 (1) 3. 7 2. 6	3. 7 . 8 (1) (1) (1) (1) (1)
son, N.Y Seattle, Wash	7. 45 7. 50	69 49	2. 9 3. 6	41 28	11 6	2. 6 1. 2	. 88 . 2	51 21	4. 2 1. 4	12 6. 7	. 05	9. 6 13	(1)

¹ Not reported.

iron plates placed in the same beaker with glass plates coated with oil base red lead paint caused a reduction in values for the glass plates of 3.4 mg. in a test period of 7 days.

Further experiments with unpainted iron plates in a very dilute solution of lead acetate caused a drop in concentration from 9.56 mg. to 1.31 mg. of lead per 500 cc. Plates painted with vinyl base paint followed by an overcoating of aluminum paint yielded as much as 0.114 mg. of lead per liter.

Water samples. For the tests of the effect of water from city supplies, samples were obtained from localities differing widely with regard to such factors as fluorides, hardness, total solids, and pH value. The normal water data given in table 1 are average results supplied by the water departments of the various cities and are not necessarily the exact mineral content of the samples used. All water samples were tested for lead by the dithizone method before use, and no lead was found.

In the experiment with oil base paint, the amount of lead found in solution after immersion for 30 days varied from 28.9 mg. per liter for water from Amarillo, Tex., to 3.62 mg. per liter for water from Seattle, Wash. The effect of the various types of water tested on the phenolic and vinyl base paints was equally variable (table 2).

Discussion

Whereas red lead purified by Glasstone's method gives somewhat lower values than those obtained with the commercial product, the value obtained by direct measurement is greater than the theoretical figure arrived at by electrode potential measurement. An explanation of the low results which Glasstone calculated from such measurements may exist in the fact $_{
m these}$ were made measurements N-NaOH (40,000 ppm). Our results show that sodium hydroxide decreases the solubility of red lead within the range investigated (fig. 7). It is possible that this may hold also for higher concentrations of sodium hydroxide.

Particularly interesting in our study are the results regarding the effect of pH and certain substances on the rate of dissolution of lead. Pertinent data are summarized in table 3.

Table 2. Dissolution of lead from red lead paint on glass plates immersed for 30 days in water from eight localities

	Milligrams of lead per liter of water per 200 cm. ²				
Locality	Oil base paint	Phenolic base paint	Vinyl base paint		
Amarillo, Tex	28. 9 27. 8 18. 2 5. 80 5. 54 4. 07 3. 62 3. 62	0. 31 . 27 . 11 . 31 1. 30 . 53 . 60 . 60	0. 17 . 03 . 18 . 38 1. 09 . 25		

They show clearly the increased rate of solution of lead during the test period in the presence of residual chlorine or at low pH.

It had been anticipated that the protective oxidized oil film, coating the particles of red lead and resulting from the drying of oil base paint, would tend to reduce the solvent action of water. Of outstanding interest, therefore, is the finding in this study of a greater amount of lead in solution following contact with red lead oil base paint in comparison with the amount resulting from red lead powder suspended in water. Thus, the theory that a protective film retards the solution of red lead is not tenable in this case.

Table 3. Solubility of red lead paint according to type of water

	Milligrams of lead per liter per 200 cm. ²				
Type of water	Oil base paint (14-day dry)	Phenolic base paint	Vinyl base paint		
Distilled	7. 4 16. 5 7. 4 4. 8 17. 5 2. 0 5. 5	0. 3 2. 4 1. 2 . 1 4. 0 . 2 . 6	0. 2 . 6 . 3 . 1 . 5 . 1		

Note: Drying times for oil base paint corrected for ease of comparison. Immersion time: 3 days.

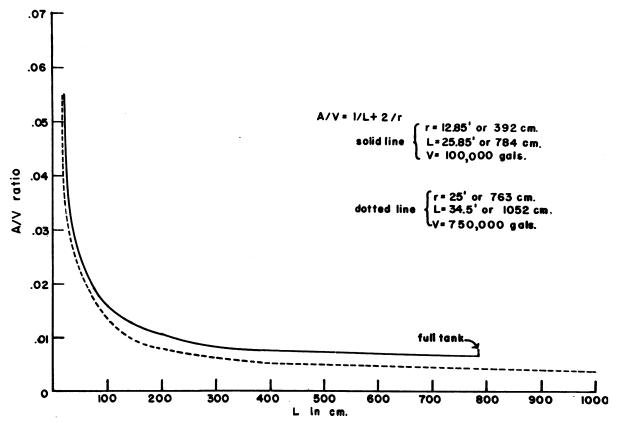
An explanation for this result is found in the effect of linseed oil on red lead itself. Red lead in contact with linseed oil results in the formation of either a small amount of lead soap, presumably lead linoleate, or a glyceride of lead (8–10). A preparation of the lead salt of the fatty acids of linseed oil exhibited solution properties similar to and values higher than the oil base film itself.

Although no figures for lead linoleate are given in the literature, values (calculated) for lead palmitate, lead myristate, lead stearate, and lead laurate (11) vary from 21.2 mg. of lead per liter of water to 46 mg. The value for the solubility of lead linoleate prepared from linseed oil used in the paint experiments in our investigation was 41.1 mg. of lead per liter of water. It is understandable, therefore, that appreciable amounts of the lead salt would leach out of this type of paint film in contact with water.

The effect of repeated leaching should provide a clue as to whether or not this was merely a surface effect yielding a relatively high initial concentration of lead, which tended to wash off and lessen in amount. The results obtained with the three types of paint exhibit such an effect. However, for the oil base paint the uptake of lead after 30 days of successive immersion approaches a nearly constant value of 2 mg. per liter per 200 cm.² of painted surface.

When the values shown in figure 3 are summed and compared with those of figure 1, it is found that, if the area of painted surface is kept constant while the volume of water is increased, the dissolution of lead from the paint surface is constant and independent of the volume. Therefore, the concentration of lead found in solution varies directly as the ratio of the area of paint to the volume of water. That this straight line relationship does not hold throughout the entire lower range of the area/

Figure 9. Area/volume ratios (cm.²/cm.³) attained in two cylindrical tanks of different dimensions as water level is increased.



Note: L=water height; r=tank radius.

volume ratio is shown by figure 4. The decrease in the amount of lead found at lower area/volume ratios may be due to the high initial solubility exhibited by the painted surfaces.

Figure 9 shows how the area/volume ratio changes with the water level in two cylindrical tanks the height of which is equal to the diameter. Comparison with figure 4 indicates that the values of lead concentration to be expected in normally operating public water supply tanks would be less than those determined experimentally with an area/volume ratio of 0.2.

Summary

The solubility of red lead powder and of lead from three types of red lead paint has been studied in a series of laboratory experiments. The effects of a number of variables were investigated separately.

The solubility of purified red lead in distilled water was found by analysis to be 8.9 x 10^{-7} gram-mol of Pb₃O₄ per liter (0.553 ppm) at room temperature.

The uptake of lead by water standing in contact with oil base red lead paint varies with the length of time of contact. In an immersion period of 6 days, 11.2 mg. of lead per liter per 200 cm.² of surface dissolved from oil base paint, 0.70 mg. from phenolic base paint, and 0.11 mg. from vinyl base paint.

The amount of lead dissolved by water in contact with oil base red lead paint varies inversely with the time of drying of the paint film.

Repeated immersion tests indicated that the leaching is partly a surface effect, and the dissolution of lead tends to approach a constant value.

Sodium hexametaphosphate causes a reduced rate of lead solvency in distilled water within the range of concentration of that used in treating public water supplies. At relatively high concentrations (greater than 15 ppm) there is an increased solubility of lead.

Sodium hydroxide up to 25 ppm depresses the rate of solution of lead with all three types of paint. An increased hydrogen-ion concentration up to pH 4 causes a pronounced increase in the rate at which lead is dissolved from the oil base as well as the phenolic and vinyl base paints.

The nature of the material painted did not affect the lead solubility provided that no free metal was in contact with the water.

Results obtained with water samples from eight localities, differing widely in chemical characteristics, indicated that such characteristics have a profound effect on the rate of solution of red lead from paint film.

Of the three paints tested, the oil base paint was at least 10 times more soluble under the conditions of the test than either the phenolic or vinyl base paints.

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